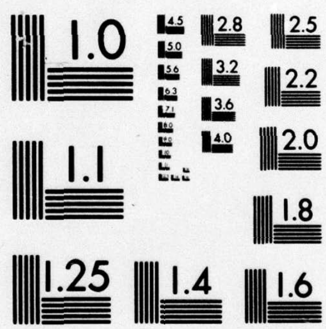
AD-A039 319 ARMY MATERIALS AND MECHANICS RESEARCH CENTER WATERTO--ETC F/G 11/10
NONDESTRUCTIVE TESTING OF RUBBER PRODUCTS USED BY THE ARMY.(U)
JAN 77 W R KLAPPERT
UNCLASSIFIED AMMRC-MS-77-1 NL

1 OF 1
AD
A039319

END
DATE
FILMED
5-77


1.0
1.1
1.25
1.4
1.6
1.8
2.0
2.2
2.5
2.8
3.2
3.6
4.0
4.5
5.0
5.6
6.3
7.1
8.0

AMMRC MS 77-1

12
NW

AD

NONDESTRUCTIVE TESTING OF RUBBER PRODUCTS USED BY THE ARMY

WALTER R. KLAPPERT
MATERIALS TESTING TECHNOLOGY DIVISION

January 1977

Approved for public release; distribution unlimited.

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

This project has been accomplished as part of the U. S. Army Materials Testing Technology Program, which has for its objective the timely establishment of testing techniques, procedures or prototype equipment (in mechanical, chemical, or nondestructive testing) to insure efficient inspection methods for materiel/material procured or maintained by DARCOM.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER
AMMRC-MS-77-1

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)
NONDESTRUCTIVE TESTING OF RUBBER PRODUCTS USED BY THE ARMY.

5. TYPE OF REPORT & PERIOD COVERED
Final Report,

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)
Walter R. Klappert

8. CONTRACT OR GRANT NUMBER(s)

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Army Materials and Mechanics Research Center
Watertown, Massachusetts 02172
AMXMR-M

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
D/A Project: PEMA
AMCMS Code: 5397.OM.6350
Agency Accession:

11. CONTROLLING OFFICE NAME AND ADDRESS
U. S. Army Materiel Development and Readiness Command
Alexandria, Virginia 22333

12. REPORT DATE
Jan [redacted]77

13. NUMBER OF PAGES
~~10~~ 12 11p.

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)

15. SECURITY CLASS. (of this report)
Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES This project has been accomplished as part of the U.S. Army Materials Testing Technology Program, which has for its objective the timely establishment of testing techniques, procedures or prototype equipment (in mechanical, chemical, or nondestructive testing) to insure efficient inspection methods for materiel/material procured or maintained by DARCOM.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)
Elastomers
Rubber
Test methods
Nondestructive testing

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

(SEE REVERSE SIDE)

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

Block No. 20

ABSTRACT

This paper is a survey of the field of rubber and rubber testing leading to a discussion of the nondestructive testing of rubber products used by the Army. It includes a primer on rubber itself, a review of physical tests, and concludes with a survey of rubber-related nondestructive testing research.

INTRODUCTION

At this time, research and development is underway to nondestructively test rubber products. This R&D is motivated by specific problems encountered by the users of rubber products. The Army, as a user of such products, has a need for the results of such research to solve problems in testing products ranging from pneumatic tires to rocket motor parts. This monograph has been prepared after a survey of the field by a member of the Nondestructive Testing Industrial Applications Branch of the Army Materials and Mechanics Research Center. It is an attempt at objectively reviewing the nondestructive testing of rubber problems. It begins with a primer on rubber itself, includes a review of physical tests, and concludes with a survey of rubber-related nondestructive testing research.

To this writer's knowledge, this is the only work of its kind and scope. Therefore, the reader is advised that this monograph is an original perception of a very complex field, and that parts of this document may be most useful as a source for productive debate. That is, several subjects in the field of rubber testing are controversial especially when vested interests are affected. However, other parts of work are easily traced back to information in well-accepted publications and can be accepted as fact. As a whole, this document should be useful as an introduction to the problems associated with the testing of rubber for those beginning to study the problem and as a good review and survey for those already involved.

A PRIMER ON ELASTOMERS, THE RUBBER COMPOUND

"Elastomer" is used as a generic term for rubber and rubber-like materials. In this report, the terms "rubber" and "elastomer" will be used interchangeably, and both will refer to the rubber compound. The polymer which is the chief ingredient in the compound will be called the "elastomeric polymer" to avoid confusion. This section briefly covers the elastomeric polymer, the filler, the curative and the other components which make up the rubber compound.

The Elastomeric Polymer

The basis of any rubber compound is the elastomeric polymer. (This polymer may be called the "elastomer" in other literature.) These polymers are long chains of molecules which vary in structure and elemental makeup according to the class of elastomeric polymer. The classes include: natural rubber, Thiokols, neoprenes, nitrile rubbers, butyl rubbers and many others. Even these classes may be divided into specific polymers and each of these polymers vary in physical properties. That is, each polymer may contribute differently to the ultimate compound with regard to inherent strength, temperature resistance, vulcanization properties and other physical and manufacturing properties. For this reason, rubber can be considered to represent a large spectrum of materials having various properties. The only common property is the ability of all rubber compounds to recover after large deformation. Finally, note that the elastomer is typically only 60 percent of the compound and there is considerable variance in this percentage from recipe to recipe.

The Filler

The filler in the rubber compound does much more than simply occupy space. The filler affects the properties of the compound. Its functions include improvement of the strength characteristics and adjustment of the cure times. Like the polymer, there is great variance in the materials used for fillers. Fillers may be pigments, fabrics, or even fibers or glass. Fabrics, fibers and other non-pigment fillers results in what may be defined as "composite materials" and this subject is considered separately in a later section.

Pigments include carbon blacks, zinc oxides, clay, calcium carbonates, and various other substances. Each of the substances is used to change the properties of the ultimate compound. Carbon blacks are used extensively in Army applications, because, in addition to their other attributes, they absorb ultraviolet light. This absorption of UV retards attack from oxygen. Fillers vary greatly in percent of compound makeup, but a typical percentage is about 30 percent of the mix.

The Curative

Unvulcanized rubber has some very poor characteristics. It is sticky when warm and rigid when cold. It is vulcanization that makes the material useful in products. For this reason, the vulcanizing agent, or curative, is a vital part of the compound. This component is required to cross-link the polymers in the vulcanization process. The cross-linked polymers cause the material to exhibit useful properties in the final product. Usually, the curative is sulfur or a peroxide type which is not used as extensively as sulfur; however, several other substances are good curatives, especially for specific applications. For example, the thiuram disulfides may be used to obtain better heat resistance. Also, certain curatives work best with certain rubbers. For example, metallic oxides work best with neoprene. In spite of the curative's importance, it is only typically 5 percent of the compound, and again, this percentage varies greatly.

The Additives

Additives comprise a relatively small percentage of the compound, usually less than 5 percent, but they are the ingredients which determine the durability and processability of the rubber material. In this report, the term "additives" includes: antioxidants, antiozonants, accelerators, activators, and all substances included under the sub-class called "softeners." As the names imply, these substances do everything from improving the rubber's resistance to the environment to making the rubber more distortable and retentive for processing. These additives are mostly organic compounds; and, though they are trace substances, it is critical that they be spread throughout a batch of rubber when it is mixed.

Mixing and Curing

The ingredients of the rubber compound are usually mixed in a two-roll rubber mill or in a Banbury internal mixer. Though both methods are considered

to give a good mix, there is no guarantee of complete dispersion of the ingredients. The mix is sampled and physical tests are performed on the sample to determine the quality of the mix. A nondestructive, complete test of the mix would be superior, but no such test exists at this time. Therefore, it is possible that failure could occur in products made from the parts of a rubber batch where components were not properly dispersed. It is also notable that a rubber product may be cured inhomogeneously. Curing takes place in the presence of heat. In fact, a rubber compound begins to cure as soon as it is mixed due to ambient heat. For this reason, there is a limited amount of time that rubber material can be stored before it is processed into a product. However, Army work has showed that peroxide-based calendered stocks can be stored at room temperature for several days or longer prior to cure without adversely affecting the properties of the vulcanizate. At some point during the production process, the temperature is increased and the rubber is vulcanized. If the rubber is vulcanized in a poorly designed mold, the heat may be spread unevenly and the material will cure irregularly. Irregular curing may also occur if the rubber product is thick because the rubber itself is a poor conductor of heat.

Another problem occurs in the curing of test pieces. Since the test piece is to represent a batch of material, it should be cured to the extent that the final product is cured. This is also difficult to guarantee.

Finally, after the rubber is vulcanized, it may continue to form cross links in the presence of ambient heat. This curing after production contributes to the aging of the end item.

THE PHYSICAL TESTING OF RUBBER

Presently, rubber products are usually tested and accepted on the basis of performance in physical tests. This is true of raw rubber materials as well as final products. Specifications for rubber products are usually written with regard to physical test performance, and even the composition of the rubber compound is left up to the manufacturer. It is admitted that the physical tests for rubber have limitations and that the rubber field is in need of improved tests. However, the parameters which could be considered in accepting rubber for a given application have caused problems to the specifiers. One result of this was a rubber developed in the 1950's which was thought to have good characteristics especially with regard to oil resistance. However, when the rubber was put into products in the field, it was found to degrade when exposed to moisture for a period of time.

In an attempt to list the problems present in the physical testing of rubber, the following list of standard tests is presented. The list is intended to survey the common tests and does not claim to include all tests, devices or standards.

The tests just listed are tests used on raw rubber materials and on samples of rubber end items. Nondestructive testing could have an advantage over these tests because it can allow inspection of 100 percent of the material or product in question. All of the physical tests listed, with the possible exception

STANDARD PHYSICAL TESTS

TEST	DEVICES	ASTM STANDARDS	PURPOSE	LIMITATIONS
Plasticity	Rotational: Mooney Viscometer Compressional: Williams Plastimeter Reciprocating motion: Monsanto Rheometer, Agfa Vulcometer, etc.	ASTM D1646, D926, D2704, D2705, D2706	Find processing characteristics of raw rubber compounds to determine scorch and cure rate.	"Results can be disappointingly variable, especially between different laboratories".[1]
Specific Gravity	Jolly Balance		To find if any gross error has been made in compounding.	Not extremely sensitive to small changes in compounding. Does not examine entire batch of raw rubber compound, only a sample.
Stress-Strain	Tension: Scott Tester, Albertoni Tester Compression: (See D575) Shear: I.C.I. slow creep test apparatus, Instron	ASTM D412, D575, D3196	To determine the strength characteristics under static load. Can be used to determine rate of cure, optimum cure, process control, acceptance testing, aging, and research.	These, along with hardness, are the most widely used tests. However, the stress on the test piece of rubber may be a poor simulation of conditions in service.
Hardness	Shore Durometer	ASTM D1415	To find hardness, the elastic modulus under small strain.	This test is only mildly destructive. However, devices are difficult to calibrate and thickness of rubber and other parameters will affect reading.
Tear	Test pieces — crescent, angle, Delft test pieces	ASTM D624	To determine tear resistance of rubber.	Tear resistance may have little to do with failure of product even if failure is due to abrasive wear. Mechanized tests are not always in agreement with hand tests.
Abrasion	Conti, Pico, and many more	ASTM D1630, 2228	To simulate some of the field conditions that the product will be subject to with respect to abrasive resistance.	Useful only in "indicating broad differences between compounds, cannot be used to discriminate closely between similar compounds".[2]
Flex Cracking	DuPont, DeMattia, Flipper, Vogt, etc.	ASTM D430-57, 813, 430-51	To evaluate rubber which will be used in high-flex applications, for example: tire sidewalls.	Poor reproducibility. Ozone may cause multiple cracking.
Fatigue	Firestone, Goodrich *Flexometers*	ASTM D623	To measure hysteresis defect by subjecting sample to complex modes of deformation.	It is notable that temperature rise is the data obtained from this test. "Practically, this measurement [hysteresis] is extremely difficult to accomplish without complications, owing to variations in the heat capacity, conductivity, and radiation of materials being tested".[3] One advantage is that complex forces are presumably closer to service conditions than bending tests.
Set, Creep, and Stress Relaxation (*Flow Properties*)	I.C.I. shear creep test apparatus	ASTM D1390, 395, 412-51T, 1206-52T	To measure the time and temperature dependent flow properties of rubber compound.	Presently, tests have poor reproducibility. "The results have little absolute meaning and are useful only for comparative or control tests".[1]
Resilience	Dunlop Pendulum, Lupke Pendulum, Goodyear-Healey Pendulum	ASTM D1054	To measure a temperature-dependent property which is analogous to the coefficient of restitution.	"Although the ... [resilience] tests give useful results, it has been pointed out that their value is limited by the fact that resilience is a function not only of the internal friction or hysteresis but also of the dynamic modulus".[2]
Low Temperature	Gehman apparatus, Kemp apparatus	ASTM D832, 797, 746, 1229, 1329, 1053	To determine the properties of rubber as it stiffens at low temperature. For example, to find the temperature at which rubber becomes brittle.	Results are extremely variable. Properties at low temperature can depend on previous history of rubber, time, and method of cooling as well as temperature.
Aging	Greer oven	ASTM D573, 572, 454, 1149, 865, 518, 1171, 1672, 2309	To determine the resistance to deterioration in an accelerated aging test by use of an air oven, oxygen pressure, ozone, light, or liquids.	In many tests, specimens may not be aged in the same oven without affecting the test. It is also difficult in some cases to assess the degree of deterioration during the test.
Resistance to Electricity, Liquids, and Gases		ASTM D257, 150, 149, D471, 1460	To determine the electrical properties or the resistance to fluids of the rubber.	Electrical properties change if there is external or internal stress. Therefore, care must be taken to assure that test piece is stressed similarly to the stress encountered in end item.
Performance Tests	Tire wheel, Road test, Aircraft tire landing simulator, other service simulators.		To determine the reaction of rubber to performance environment.	Tests are generally costly and destructive. Obviously, only a sample of the end items can be tested. Furthermore, simulators rarely replicate all of the performance variables needed for complete evaluation.

1. BLOW, C. M. *Rubber Technology and Manufacture.* Butterworth, London, 1971.
2. NAUNTON, ed. *The Applied Science of Rubber.* E. Arnold, London, 1961.
3. MORTON, M. *Introduction to Rubber Technology.* 2d ed., Van Nostrand Reinhold, New York, 1973.

of hardness, destroy or seriously degrade the part of the rubber being tested. Therefore, these tests are limited to the testing of samples or coupons. Granted, it is generally difficult to conceive of a nondestructive test which would give the same information as a particular physical test. However, the creation of such nondestructive tests is a goal which needs to be considered.

RUBBER IN CONJUNCTION WITH OTHER MATERIALS

In many items, rubber is used with other materials. For example, textiles and fibers are used as fillers in the rubber compound to produce materials used in tent materials, belt materials and in other items where strength and/or liquid impermeability are required. Rubber may also be bonded (usually "vulcanize" bonded) to other materials, typically to metal or textiles. This may be used to reduce noise or avoid destruction by the metal item, or to protect the metal or textile from the environment. Applications include Army tank treads and roadwheels as well as rubber coatings for handweapons. The bonding of rubber to textiles also becomes important in tire manufacture. However, the tire is such a complex rubber product that it might be best classified as a composite of rubber and non-rubber items rather than a compound product. Tire testing is a full study in itself.

The tests on products which contain rubber and other items are chiefly performance tests. However, the greatest amount of nondestructive testing research has been done on these compound items and a few physical tests are used to determine the strength-of-bond. Two of these physical tests follow:

BONDING TESTS

TEST	DEVICE	ASTM STDS	PURPOSE	LIMITATIONS
Rubber-to-Textile Adhesion	Peel devices dead load, H-block	D413 D2138	Determine Adhesion	Stress on item does not replicate stress in operation.
Rubber-to-Metal	Peel devices plate test specimens, conical test specimens, shear tests	D429 D2229	Determine Adhesion	Stress on item does not replicate stress in operation.

The tests listed above are static tests. That is, the bond is tested under a static load. Dynamic tests are being developed at this time.

NDT OF RUBBER ITEMS

Most work done in nondestructively testing rubber has been work done in inspecting end items rather than raw materials. In many cases, the NDT is applied without full information on the failure modes of the item being inspected. There may simply be a hope that a nondestructive test will be found that

correlates with performance of the item. Though this may not be a rigorous approach, the NDT test, once proven, may be superior to a destructive physical test. Physical tests may be based on more basic material characteristics, but the destructive measurement of these characteristics may not correlate well with performance.

The principal approaches which have been applied to the nondestructive testing of rubber are: ultrasonics, radiography, optical holography and thermal methods. Generally, all of these approaches can be tried in the attempt to solve a rubber problem, but the approaches will yield different information which will be more or less relevant to the problem. For example, when inspecting an automobile tire for separations between ply layers, the inspector will find that both optical holography and ultrasonic methods will be useful. But radiography will probably be useless. On the other hand, if one is inspecting for broken and misplaced cords (particularly steel cords), then radiographic methods are most useful and the other methods become comparatively useless. In general, if the anomaly which leads to failure is known, an NDT approach may be able to detect it. However, the anomaly which leads to failure usually is not known or is the subject of debate. Therefore, research has tended to examine tires (and other rubber products) by some NDT approach and look for correlation between results and field tests. It has become common to examine for anomalies called by some generic name like "degradation" or "structural integrity."

The lack of information on failure modes extends beyond examination of the pneumatic tires. Tank treads and roadwheels, though simpler than tires, also have debatable failure modes. The anomaly usually inspected for in the tank treads and roadwheels is an unbonded area between the steel and rubber parts of the product. Certainly such a debond could lead to failure, but it is argued that the items also fail due to environmental attack and misuse. Again, the recourse has been to test NDT results against field test results. NDT has also been applied to the inspection of elastomeric rocket motor case liners in an attempt to find separations. However, from the information available to this writer, the work on case liners is still highly experimental.

In summary, many standard NDT approaches (such as radiography and ultrasonics) can be applied to rubber product problems. Also, newer approaches (such as thermal methods and optical holography) can be developed to enhance on standard approach results or to obtain new information altogether. The great gap lies in that the NDT inspector usually does not know what anomaly he should try to find. But, in spite of this unknown, some NDT work in rubber goes on in the hope of finding an inspection method from statistical correlation rather than a rigorous approach.

SUMMARY

This report has been an introduction to the problems associated with the nondestructive testing of elastomeric products. It has reviewed the basics of rubber compounding and physical testing and has briefly surveyed the problem in the Army concerning elastomeric products.

BIBLIOGRAPHY

ALBERTSON, *Application of Neutron Radiography Using a Radioactive Neutron Source*, General Electric, 1969.

Automobile Tire Hot-Spot Detection, Barnes Engineering Company.

BIBBY, *Nondestructive Infrared Tire Testing*, Monsanto, 1972.

BLOW, *Rubber Technology and Manufacture*, Butterworth, London, 1971.

BOBO, *An Infrared Experiment on a Road Wheel During an FMVSS 109 Type Compliance Test*, National Highway Traffic Safety Administration, 1972.

BOBO, *Isolation of Flaws by Use of Thermal Differentials on a Tire Under Mild Loading Conditions*, DOT, Cambridge, 1972.

BOUTAINE, *A Radiographic Tracer Method for NDT of Aircraft Tires*, A.T.A. Nondestructive Testing Forum, 1974.

COLLINS, *Solid Rocket Motor Inspection by Acoustical Holography Techniques*, Air Force Rocket Propulsion Laboratory, 1975.

DEAN, *Air-Coupled Ultrasonic Probe*, British Journal of NDT, 1968.

FRANING, *Test Report M140 Gun Mount Seals*, ARMCOM.

FRERSON, *Holographic Interferometry Techniques and Applications*, ARMCOM, 1969.

GARDNER, *Investigation of Techniques for the Development of a Seam Inspection Method for Flexible Fuel Storage Container Material*, Southwest Research, 1970.

HALSEY, *The Nondestructive Testing of Passenger Tires*, Scientific Testing Laboratory, Materials Evaluation, Indiana, Pennsylvania, 1968.

HENDERSON, *Radiographic Techniques for Examining the Filament-Wound Tire*, Wright-Patterson AFB, 1968.

IVERSON, *Holographic-Nondestructive Testing of Rubber-to-Metal Bonds*, ARMCOM, 1969.

JACOBY, *Microwave Measurement of Aerospace Materials*, Lockheed Missiles and Space Company, 1972.

LAVERY, *Nondestructive Tire Testing Studies*, DOT, Cambridge, 1972.

LEVINE, *Dynamic Infrared Inspection Techniques*, GATX, 1963.

LICHODZIEJEWSKI, *Nondestructive Inspection of Track Shoe Rubber Pads*, GATX, International Journal of NDT, 1972.

MARTIN, *The Application of a Radio Influence of Voltage Test to the Detection of Voids in Elastomers*, Materials Evaluation, 1970.

MORTON, *Introduction to Rubber Technology*, 2d ed., Van Nostrand Reinhold, New York, 1973.

NAUNTON, *The Applied Science of Rubber*, E. Arnold, London, 1961.

PRUSINSKI, *Bond-Line Inspection Probe for Rubber-Steel Composites*, GATX, 1972.

Rubber in Engineering, Chemical Publishing Company, Brooklyn, New York, 1946.

SPERRY, *A New Generation of Aircraft Tire Test Dynamometer Systems*, DoD Conference on NDT, 1970.

VOGEL, *Proceedings of the First, Second and Third Symposia on the NDT of Tires*, Army Materials and Mechanics Research Center, 1973-1976.

WALTERS, *Failure Analysis of Wire-Reinforced Flex Hose Used in Damping Retract/ Reconnect System*, NASA/Kennedy Space Center, 1973.

WILLIAMS, *Environmental Exposure and Accelerated Testing of Rubber-to-Metal Vulcanize Bonded Assemblies*, ARMCOM, 1975.